

TRANSMITTAL LETTER TO THE UNITED STATES

DESIGNATED/ELECTED OFFICE (DO/EO/US)

CONCERNING A FILING UNDER 35 U.S.C. 371

07089.0010U1

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.5)

09/743528

INTERNATIONAL APPLICATION NO

PCT/EP98/04563

INTERNATIONAL FILING DATE

21 July 1998

PRIORITY DATE CLAIMED

TITLE OF INVENTION

SYNTHESIS GAS PRODUCTION BY STEAM REFORMING

APPLICANT(S) FOR DO/EO/US

Dybkaer, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 18 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
A **SECOND** or **SUBSEQUENT** preliminary amendment.
16. ☐ A substitute specification.
17. ☐ A change of power of attorney and/or address letter.
18. ☒ Certificate of Mailing by Express Mail
19. ☒ Other items or information:

Return Postcard

Authorization to Treat Reply Requiring Extension of Time as Incorporating Petition for Extension of Time

EL491586646US

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.5) 09/743528		INTERNATIONAL APPLICATION NO. PCT/EP98/04563		ATTORNEY'S DOCKET NUMBER 07089.0010U1	
20. The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :				CALCULATIONS PTO USE ONLY	
<input checked="" type="checkbox"/> Search Report has been prepared by the EPO or JPO \$840.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) \$670.00 <input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$760.00 <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$970.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$96.00					
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$860.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).				\$130.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	2 - 20 =	0	x \$18.00	\$0.00	
Independent claims	1 - 3 =	0	x \$80.00	\$0.00	
Multiple Dependent Claims (check if applicable). <input type="checkbox"/>				\$0.00	
TOTAL OF ABOVE CALCULATIONS =				\$990.00	
Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable). <input type="checkbox"/>				\$0.00	
SUBTOTAL =				\$990.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492 (f)).				\$0.00	
TOTAL NATIONAL FEE =				\$990.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input type="checkbox"/>				\$0.00	
TOTAL FEES ENCLOSED =				\$990.00	
				Amount to be:	\$
				refunded	
				charged	\$

- ☒ A check in the amount of **\$990.00** to cover the above fees is enclosed.
- ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **14-0629** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Attn: Mitchell A. Katz, Esq.
NEEDLE & ROSENBERG, P.C.
127 Peachtree Street, N.E., Suite 1200
Atlanta, GA 30303, US
404-688-0770

Mitchell A. Katz
SIGNATURE

Mitchell A. Katz

NAME

33,919

REGISTRATION NUMBER


10 January 2001
DATE

09/743528

JC04 Rec'd PCT/PTO 1 0 JAN 2001

CERTIFICATE OF EXPRESS MAILING

I hereby certify that the U.S. National Phase Application in the name of: Dybkjaer *et al.* consisting, of: Transmittal Letter to the United States Designated/Elected Office (DO/EO/US) Concerning a Filing Under 35 U.S.C. 371 PTO Form 1390 (2 Pages); a First Preliminary Amendment; an Authorization to Treat Reply Requiring Extension of Time as Incorporating Petition for Extension of Time; a copy of the International Search Report; a copy of the International Preliminary Examination Report; a Check in the amount of \$990.00 to cover filing fees; and a return postcard for "SYNTHESIS GAS PRODUCTION BY STEAM REFORMING" are being deposited with the United States Postal Service as Express Mailing No. EL491586646US in an envelope addressed to: Assistant Commissioner for Patents, Box PCT (IPEA/EP), Washington, D.C. 20231, on this **10th** day of **JANUARY 2001**.



EVERARDO MCFARLANE

1-10-2001

Date

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09/743528

JC07 Rec'd PCT/PTO 10 JAN 2001

ATTORNEY DOCKET NO. 07089.0010U1
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of)
)
Dybkjaer et al.) Group Art Unit: Unassigned
)
Serial No.: Unassigned as yet) Examiner: Unassigned
(35 U.S.C. 371 application from)
PCT/EP98/04563))
Filed: Herewith)
)
For: "SYNTHESIS GAS PRODUCTION BY)
STEAM REFORMING")

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
BOX PCT (ISA/EP)
Washington, D.C. 20231

NEEDLE & ROSENBERG, P.C.
Suite 1200, The Candler Building
127 Peachtree Street, N.E.
Atlanta, Georgia 30303-1811

January 10, 2001

Sir:

This Preliminary Amendment is being filed concurrently with the above-captioned U.S. National Phase Application under 35 U.S.C. 371. Prior to examining the above-identified patent application, please amend the claims of the application, as amended in the preliminary examination under Article 34, as follows and consider the following remarks.

IN THE CLAIMS

Please amend claim 1, as follows:

1. (Amended) Process for the preparation of hydrogen and carbon monoxide rich gas by steam reforming of a hydrocarbon feedstock in the presence of a steam reforming catalyst supported as thin film on the wall of a reactor, comprising the steps of
- (a) [optionally] passing a process gas of hydrocarbon feedstock through a first reactor (10) with a thin film of steam reforming catalyst supported on walls of the reactor (10) in a heat conducting relationship with a hot gas stream of flue gas;
 - (b) passing effluent from the first reactor (10) to a subsequent tubular reactor (14) being provided with a thin film of steam reforming catalyst and/or steam reforming catalyst pellets and being heated by burning of fuel, thereby obtaining a partially steam reformed gas effluent and [a] the hot gas stream of flue gas;
 - (c) passing the effluent from the second reactor (14) to an autothermal reformer (16); and
 - (d) withdrawing from the autothermal reformer (16) a hot gas stream of product gas (20) rich in hydrogen and carbon monoxide.

REMARKS

Claims 1 and 2 are pending in this application. Claim 2 remains unamended from its original form. Please examine claim 1, as amended, and claim 2.

The Amended Sheet annexed to the IPER of 27 September 1999, only contained claim 1 and did not repeat claim 2 (claim 2 was unamended). In an abundance of caution, applicants are submitting claim 1 as amended via this preliminary amendment rather than via the IPER annex so that both claims 1 and 2 are assured to be considered for examination.

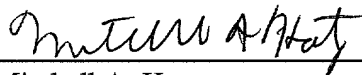
ATTORNEY DOCKET NO. 07089.0010U1

Pursuant to the above amendments and remarks, consideration and allowance of the pending application is believed to be warranted. The Examiner is invited and encouraged to directly contact the undersigned if such contact may enhance the efficient prosecution of this application to issue.

No additional fee for this amendment is believed due. However, the Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 14-0629.

Respectfully submitted,

NEEDLE & ROSENBERG, P.C.



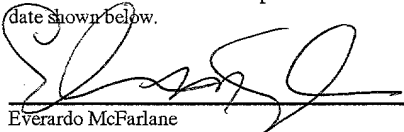
Mitchell A. Katz

Registration No. 33,919

Needle & Rosenberg, P.C.
Suite 1200, The Candler Building
127 Peachtree Street, N.E.
Atlanta, Georgia 30303-1811
(404) 688-0770 (Telephone)
(404) 688-9880 (Fax)

CERTIFICATE OF EXPRESS MAIL

I hereby certify that this correspondence is being deposited with the United States Postal Service as Express Mail Certificate No. EL491586646US in an envelope addressed to: Assistant Commissioner for Patents, BOX PCT (ISA/EP), Washington, D.C. 20231, on the date shown below.


Everardo McFarlane

1-10-01
Date

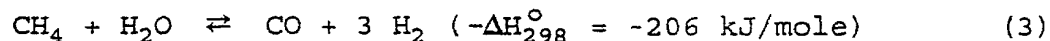
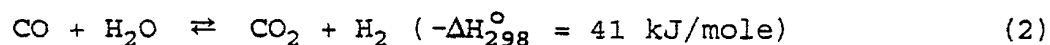
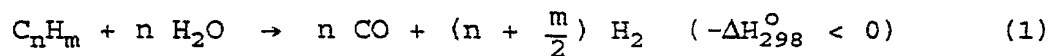
Synthesis Gas Production by Steam Reforming

The present invention is directed to the production of synthesis gas by steam reforming of a hydrocarbon feedstock in contact with catalyzed hardware.

The term catalyzed hardware is used for a catalyst system, where a layer of catalyst is fixed on a surface of another material, e.g. metallic surfaces. The other material serves as the supporting structure giving strength to the system. This allows to design catalyst shapes which would not have sufficient mechanical strength in itself. The system herein consists of tubes on which a thin layer of reforming catalyst is placed on the inner wall.

Alternative layouts may comprise of tubes with a catalyst layer on the outside, plates with catalyst coating, or other suitable shapes.

Synthesis gas is produced from hydrocarbons by steam reforming by the reactions (1)-(3):



A second method for production of synthesis gas is autothermal reforming (ATR). In autothermal reforming, combustion of hydrocarbon feed is carried out with substoichiometric amounts of oxygen by flame reactions in a burner combustion zone and, subsequently, steam reforming of the partially combusted feedstock in a fixed bed of steam reforming catalyst. The oxidant can be air, enriched air, or pure oxygen.

A third method for production of synthesis gas is the combination of first passing the hydrocarbon feed through a fixed bed of reforming catalyst and, subsequently, passing the partly reformed feed through an

autothermal reformer. The fixed bed may comprise of a number of tubes placed in a fired furnace. This combination is called two-step reforming or primary followed by secondary reforming and is particularly suited for production of synthesis gas for methanol and ammonia production. By controlling the amount of reforming occurring in the fixed bed steam reformer before the ATR, a synthesis gas having the correct stoichiometry for methanol synthesis or a synthesis gas having the correct ratio of hydrogen to nitrogen for ammonia synthesis can be produced.

State of the art steam reforming technology makes use of reforming catalyst in the form of pellets of various sizes and shapes. The catalyst pellets are placed in fixed bed reactors (reformer tubes). The reforming reaction is endothermic. In conventional reformers, the necessary heat for the reaction is supplied from the environment outside the tubes usually by a combination of radiation and convection to the outer side of the reformer tube. The heat is transferred to the inner side of the tube by heat conduction through the tube wall and is transferred to the gas phase by convection. Finally, the heat is transferred from the gas phase to the catalyst pellet by convection. The catalyst temperature can be more than 100°C lower than the inner tube wall temperature at the same axial position of the reformer tube.

It has been found that heat transport is more efficient when catalyzed hardware is used in the steam reforming process. The heat transport to the catalyst occurs by conduction from the inner tube wall. This is a much more efficient transport mechanism than the transport by convection via the gas phase. The result is that the temperatures of the inner tube wall and the catalyst are almost identical (the difference below 5°C). Furthermore, the tube thickness can be reduced, see below, which makes the temperature difference between the inner and outer side of the reformer tube smaller. It is hence possible to have

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both a higher catalyst temperature and a lower tube temperature, all other conditions being the same when replacing the conventional reformer tubes with catalyzed hardware tubes. A low outer tube wall temperature is desirable since it prolongs the lifetime of the tube. A high catalyst temperature is advantageous since the reaction rate increases with temperature and since the equilibrium of reaction (3) is shifted to the right hand side resulting in a better utilisation of the feed.

Pressure drop in the catalyzed reformer tube is much lower than in the conventional case for the same tube diameter. This enables the use of reactors of non-traditional shapes e.g. tubes with small diameter and still maintaining an acceptable pressure drop. Smaller tube diameter results in an increased tube lifetime, tolerates higher temperatures and reduces the tube material consumption.

Finally, the catalyst amount is reduced when using catalyzed hardware reformer tubes compared to the conventional reformer with a fixed bed of reforming catalyst.

The small amount of catalyst dictates the use of a feedstock free of catalyst poisons. This can e.g. be obtained by sending the feedstock through a prereformer.

Fig. 1 shows an example of a plant producing syn-gas. Feed 2 is preheated, desulphurized in unit 4, mixed with process steam 6, and further heated before entering an adiabatic prereformer 8. The effluent stream from prereformer 8 is further heated in a heat exchanger coil arranged in flue gas channel 12 and send to the tubular reformer 14, where conversion of methane to hydrogen, carbon monoxide, and carbon dioxide occurs. The effluent gas is passed to autothermal reformer 16, wherein combustion is performed with oxidant stream 18. The processing of effluent gas 20 downstream from the autothermal reformer depends on the intended use of the product.

Catalyzed hardware can be used in two of the units shown in Fig. 1:

1. In the preheater coil 10 for heating the prereformer effluent gas before entering the tubular reformer 14.
2. In the tubular reformer 14.

This invention provides process for the preparation of hydrogen and carbon monoxide rich gas by steam reforming of a hydrocarbon feedstock in presence of a steam reforming catalyst supported as thin film on the wall of a reactor, comprising steps of

- (a) optionally passing a process gas of hydrocarbon feedstock through a first reactor with a thin film of steam reforming catalyst supported on walls of the reactor in heat conducting relationship with a hot gas stream;
- (b) passing effluent from the first reactor to a subsequent tubular reactor being provided with a thin film of steam reforming catalyst and/or steam reforming catalyst pellets and being heated by burning of fuel, thereby obtaining a partially steam reformed gas effluent and a hot gas stream of flue gas;
- (c) passing the effluent from the second reactor to an autothermal reformer; and
- (d) withdrawing from the autothermal reformer a hot gas stream of product gas rich in hydrogen and carbon monoxide.

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Example 1

A catalyzed hardware reformer reactor has been tested. The test unit consists of a system for providing the feeds to the reactor, the reactor itself, and equipment for posttreatment and analysis of the effluent gas from the reactor.

The reactor consists of a 1/4" tube of length 1050 mm which is, in the middle 500 mm, coated on the inner wall with RKNR nickel steam reforming catalyst. The catalyst has the same composition as the RKNR pelletshaped steam reforming catalyst available from Haldor Topsoe A/S. The thickness of the catalyst layer is 0.31 mm. The catalyzed reactor tube is placed in a casing made of solid metal, which has a hole closing tightly around the catalyzed tube. A number of milled grooves, in which thermocouples are placed, is made along the hole. One of the thermocouples is movable so that a wall temperature profile of the catalyzed tube can be obtained. Additionally, thermocouples are placed in the gas channel in the catalyzed tube measuring the gas temperature at the inlet to and the outlet from the catalyzed zone. The reactor with casing is placed in an electrically heated oven, in which the temperature can be controlled separately in 6 different zones.

The feed steams consist of hydrogen, methane, carbondioxide, and steam. The feed streams are mixed and preheated before entering the reactor. After the reactor, the effluent gas stream is cooled down, the condensed water is separated from the gas, and the gas composition is measured by a gaschromatograph.

Two sets of conditions were tested. One set at lower temperature to simulate use of catalyzed hardware in a preheater coil (test No. 1), and one set at higher temperature to simulate a tubular reformer (test No. 2). The conditions are shown in Table 1. The pressure was in both cases 28 bar g. The temperature profile imposed on the

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reactor wall measured by the movable thermocouple is shown in Fig. 2.

Table 1

Conditions for Catalyzed Hardware Reactor Test						
Test No.	Gas temperature at inlet of catalyzed zone	Gas temperature at outlet of catalyzed zone	Hydrogen flow rate	Methane flow rate	Carbon-dioxide flow rate	Steam flow rate
	°C	°C	Nl/h	Nl/h	Nl/h	Nl/h
1	605	633	62.0	310.0	16.1	781.4
2	679	795	240.5	152.0	63.1	425.0

The measured effluent gas composition is shown in Table 2. The gas composition is on dry basis.

Table 2

Effluent Gas Composition on Dry Basis				
Test No.	Hydrogen	Carbon-monoxide	Carbon-dioxide	Methane
	mole%	mole%	mole%	mole%
1	49.6	2.48	11.4	36.5
2	67.8	10.8	9.80	11.7

The effluent gas is in both cases in equilibrium with respect to the reforming reaction at the outlet gas temperature within experimental uncertainty. This demonstrates that a conversion similar to a fixed bed reactor can be obtained in a catalyzed hardware reactor.

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CLAIMS

1. Process for the preparation of hydrogen and carbon monoxide rich gas by steam reforming of a hydrocarbon feedstock in presence of a steam reforming catalyst supported as thin film on the wall of a reactor, comprising steps of
- (a) optionally passing a process gas of hydrocarbon feedstock through a first reactor with a thin film of steam reforming catalyst supported on walls of the reactor in heat conducting relationship with a hot gas stream;
 - (b) passing effluent from the first reactor to a subsequent tubular reactor being provided with a thin film of steam reforming catalyst and/or steam reforming catalyst pellets and being heated by burning of fuel, thereby obtaining a partially steam reformed gas effluent and a hot gas stream of flue gas;
 - (c) passing the effluent from the second reactor to an autothermal reformer; and
 - (d) withdrawing from the autothermal reformer a hot gas stream of product gas rich in hydrogen and carbon monoxide.
2. Process of claim 1, wherein the steam reforming catalyst comprises nickel and/or noble metals.

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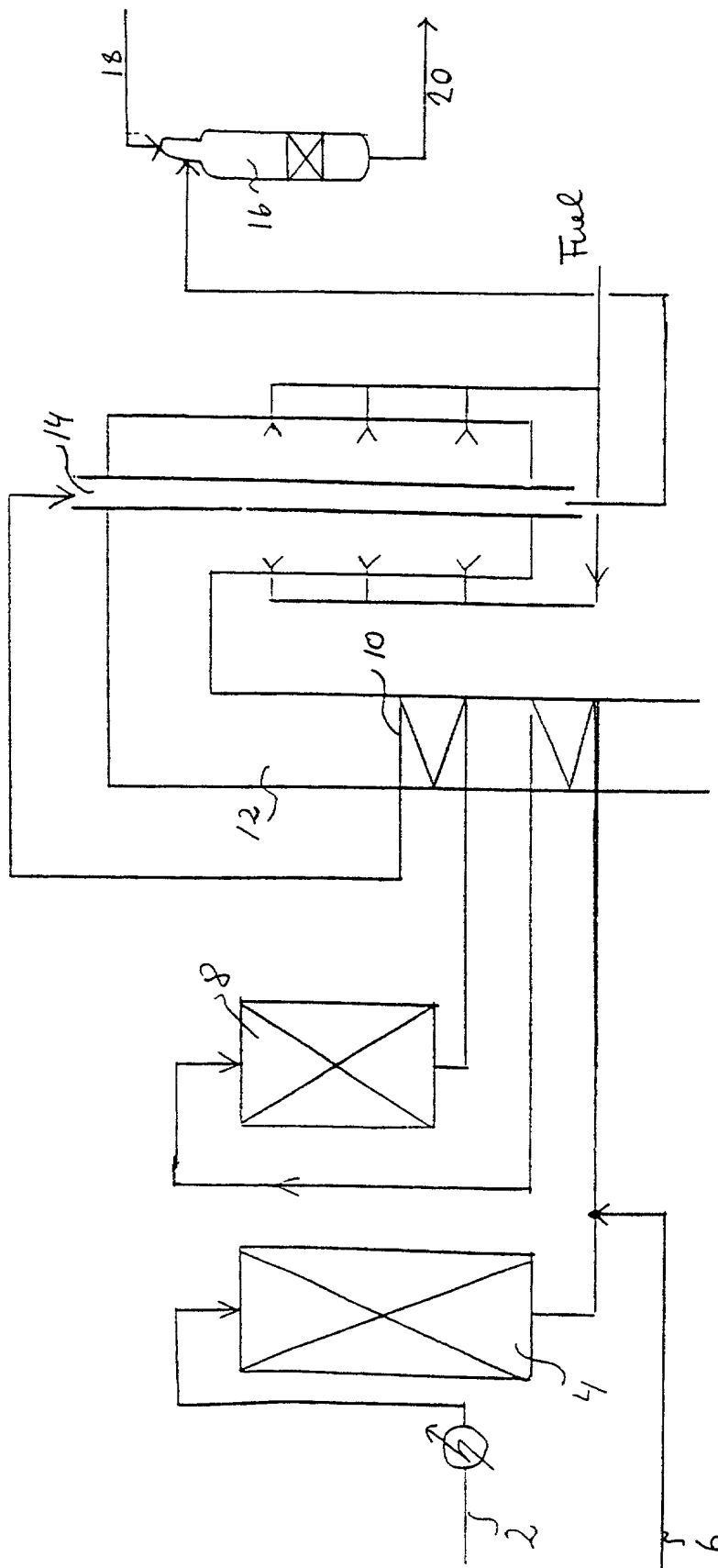


FIG. 1

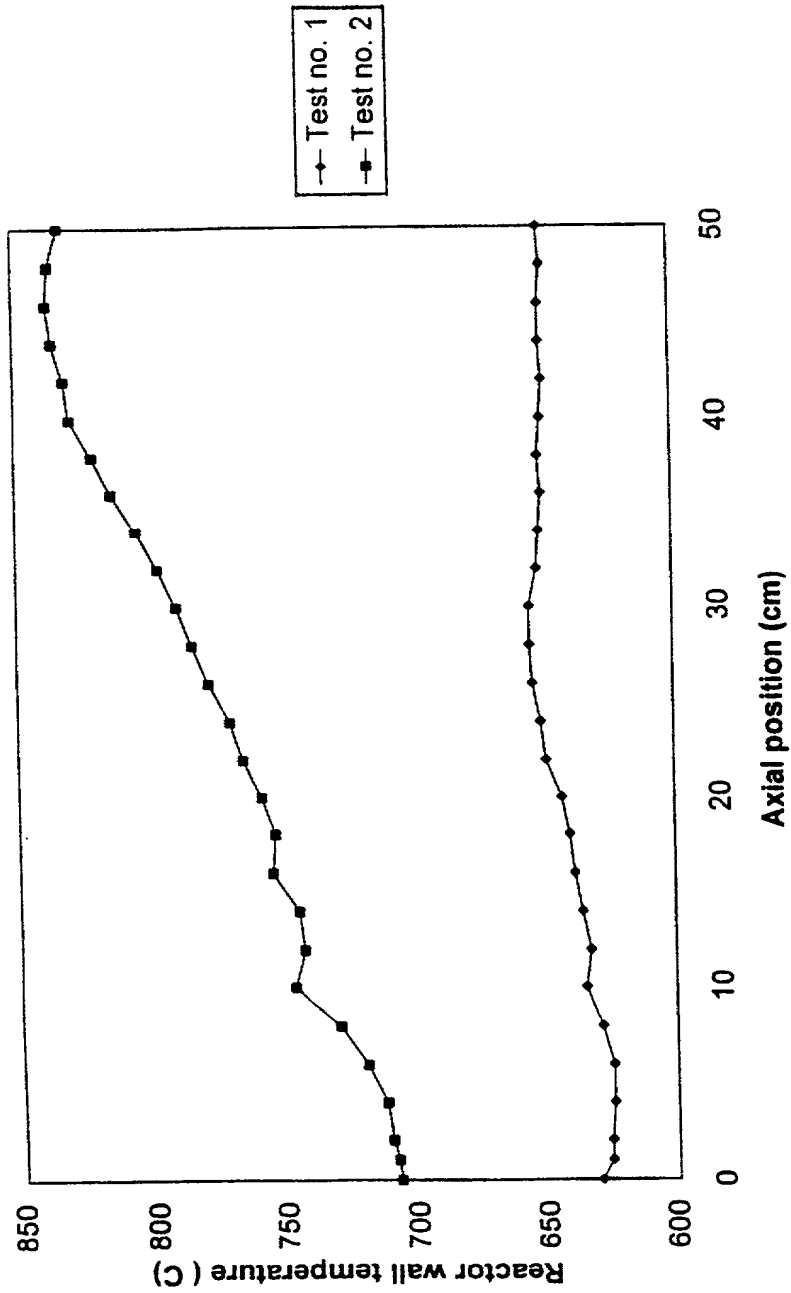


FIG. 2

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

(X) Original () Supplemental () Substitute () PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled "**SYNTHESIS GAS PRODUCTION BY STEAM REFORMING**", which is described and claimed in the specification

(check one) ☐ which is attached hereto, or
 ☐ which was filed on , as United States Application No. and with amendments through (if applicable), or
 ☒ in International Application No. PCT/EP98/04563, filed July 21, 1998, and as amended on September 8, 1999, and filed in the U.S. under 35 U.S.C. 371 on January 10, 2001, and with Preliminary Amendment filed concurrently on January 10, 2001.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information known by me to be material to the patentability of the claims of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119 (a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) or §365(b) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATIONS: (ENTER BELOW IF APPLICABLE)			PRIORITY CLAIMED (MARK APPROPRIATE BOX BELOW)	
APP. NUMBER	COUNTRY	DAY/MONTH/YEAR FILED	YES	NO
PCT/EP98/04563	PCT	21 July 1998	X	

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below.

APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose all information known by me to be material to the patentability of the claims of this application as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NO.	FILING DATE	STATUS (MARK APPROPRIATE COLUMN BELOW)		
		PATENTED	PENDING	ABANDONED

I hereby appoint the following attorneys and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:



Address all telephone calls to Mitchell A. Katz at telephone no. (404) 688-0770.

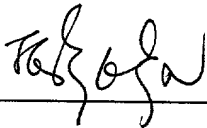
Address all correspondence to:

Mitchell A. Katz
NEEDLE & ROSENBERG, P.C.
Suite 1200, The Candler Building
127 Peachtree Street, N.E.
Atlanta, Georgia 30303-1811

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00
Full name of first inventor:Ib Dybkjaer

Inventor's signature:

Date: 08 March 2001

Residence:

Ndr. Frihavnsgade 25, 3.tv, DK 2100 Copenhagen Ø, Denmark

Post Office Address:

Ndr. Frihavnsgade 25, 3.tv, DK 2100 Copenhagen Ø, Denmark

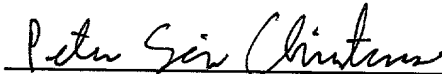
Citizenship:

Denmark

DKX

2-00
Full name of second inventor:Peter Seier Christensen

Inventor's signature:

Date: 08 March 2001

Residence:

Glasvej 7, 1.tv., DK 2400 Copenhagen NV, Denmark

Post Office Address:

Glasvej 7, 1.tv., DK 2400 Copenhagen NV, Denmark

Citizenship:

Denmark

DKX

3-00
Full name of third inventor:Viggo Lucassen Hansen

Inventor's signature:

Date: 08 March 2001

Residence:

Botoftevej 12, DK-2700 Brønshøj, Denmark

Post Office Address:

Botoftevej 12, DK-2700 Brønshøj, Denmark

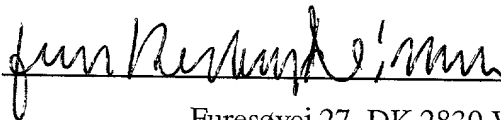
Citizenship:

Denmark

DKX

4-00
Full name of fourth inventor:J.R. Rostrup-Nielsen

Inventor's signature:

Date: 08 March 2001

Residence:

Furesøvej 27, DK 2830 Virum, Denmark

Post Office Address:

Furesøvej 27, DK 2830 Virum, Denmark

Citizenship:

Denmark

DKX